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Oilseed Standards

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Report Highlights:
On July 3, 2008, China notified the WTO of the National Standard GB-1532-2006 "National Standard for Soybeans" as TBT/N/CHN/402. This standard specifies the relevant terms and definitions, classifications, quality requirements, test methods, and requirements for labeling, packaging, transportation and storage of soybeans. GB/T 5511 Inspection of Grain and Oilseeds - Methods for Determination of Crude Protein is referenced in that standard and published here as a reference in reviewing TBT/N/CHN/402. This report is an UNOFFICIAL translation of GB/T 5511.

Includes PSD Changes: No
Includes Trade Matrix: No
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[CH]

Executive Summary: On July 3, 2008, China notified the WTO of the National Standard GB-1532-2006 "National Standard for Soybeans" (Replacing GB 1352-1986) as TBT/N/CHN/402. This standard specifies the relevant terms and definitions, classifications, quality requirements, test methods, and requirements for labeling, packaging, transportation and storage of soybeans. This standard also applies to testing, evaluation and identification of the quality of commercial soybeans. The date for submission of final comments to the WTO is September 3, 2008. The proposed date of adoption is 90 days after circulation by the WTO Secretariat (October 3, 2008) and the proposed date of entry into force is 6 months after adoption (January 3, 2009). This is notified as GAIN Report CH8066.

One of the measures that is referenced in the proposed National Standard is GB/T 5511 Inspection of Grain and Oilseeds - Methods for Determination of Crude Protein. This standard has not been notified to the WTO. This National Standard, along with other standards published in GAIN Reports CH8097-CH8105, is being published so that GB-1532-2006 "National Standard for Soybeans" TBT/N/CHN/402 can be reviewed with this additional pertinent information.

Thanks go to the United States Soybean Export Council – International Marketing and the U.S. Grains Council for their support in translating this measure.

BEGIN TRANSLATION

National Standard of the People's Republic of China

GB 5511-85

Inspection of Grain and Oilseeds - Methods for Determination of Crude Protein

Issued on Nov. 2, 1985 Implemented on July 1, 1986

This standard is applicable to determination of crude protein in commodity grain and oilseeds.

1 Instruments and Apparatus

1.1 Kjeldahl flask: 50ml;

1.2 Round-bottom flask: 1000ml;

1.3 Wire coil heater;

1.4 Conical flask: 100ml;

1.5 Microburette: 5ml, scale 0.01ml;

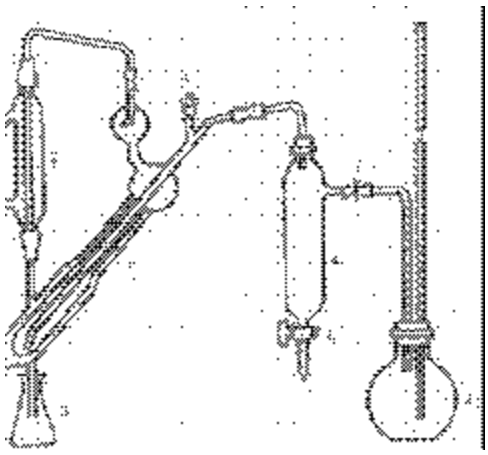
1.6 Volumetric flasks: 50ml;

1.7 Pipette: 5ml

1.8 Graduated flask: 10ml;

1.9 Rubber suction bulb;

1.10 Micro-kjeldahl distillation apparatus (see the figure below), rubber pipe etc.



Micro-kjeldahl Distillation Apparatus

1-Matrass; 2-Condensate tube; 3-Adopter flask; 4-Backflow tube; 5-Funnel; 6-Piston; 7-Pinchcock; 8-Flask

2 Reagents

2.1 Concentrated sulfuric acid-hydrogen peroxide-water mixed solution (2:1:3): Add 200ml concentrated sulfuric acid gradually into 100ml water, cool the mixture and add 30% hydrogen peroxide (300ml), and then mix evenly for stand-by. The mixture can be preserved for a month in a cool place.

2.2 Mixed catalyzer: Collect 10g copper sulphate ($CuSO_4 \cdot 5H_2O$), 100g potassium sulfate (A.R), and 0.2g selenium powder, porphyrize them in a mortar so that they can pass the 40-mesh sieve, and finally mix them evenly for stand-by.

2.3 40% sodium hydroxide solution;

2.4 2% boric acid solution;

2.5 0.01N Hydrochloric acid solutions.

2.6 Methyl red ethanol solution: Dissolve 0.1g methyl red in 75g 95% ethanol (first grind the ethanol in the mortar);

2.7 Methylene blue ethanol solution: Dissolve 0.1g methylene in 80ml 95% ethanol. Before using, mix the two said solutions in a proportion of 2:1. It appears violet red in acid area, achromatic color when PH is 5.5, and green in alkali area.

3 Operation Method

3.1 Digestion

The quantity of test sample is calculated based on the crude protein content of 10~30mg. Generally, weigh up test sample of 0.2~0.3g (*W*, accurate to ±0.0001g), roll it with flint-glazed paper, pour it into a 50ml kjeldahl flask, and then add 1g mixed indicator together with 3-5ml sulfuric acid-hydrogen peroxide-water mixed solutions. Shake it gently and slant it on the electric heater, heat it for digestion in a fume cupboard. Heat it at a low temperature at the beginning, during which period the foam in the flask is not allowed to overrun 2/3 of the flask belly. After the foam decreases and the smoke becomes white, increase the temperature to keep it boiling gently. When the solution is digested and appears light blue-green transparency, keep heating to boil it for 10 minutes. Take out the solution to be digested, cool it at the room-temperature, and then add 10-20ml water.

After the solution decreases to the room-temperature, cleanly pour it into a 50ml volumetric flask. Add water to dilute it to the scale and mix for stand-by.

3.2 Distillation

Distillation and imbibing are implemented according to setup of the fig.: Micro-kjeldahl Distillation Apparatus.

Add 400ml water and some small broken sheet glass in No. 8 flask, then successively add 5 drops of mixed indicator and some drops of acid until the solution appears violet red. Connect No. 4, 1, 2 flasks and check if they are airtight.

Measure and take 30ml 1% boracic acid solution and pour it into No.3 conical flask, add 2 drops of mixed indicator and insert the condenser tube underneath 1cm of the solution level. Measure and take 5ml dissolved digestion solution, pour it into No.1 flask with No.5 funnel, and then add 10ml water to wash No.5 funnel. Weigh 1ml 40% sodium hydroxide solution, lift the piston and pour the solution into No.1 flask, wash No.5 funnel with 2-5ml water immediately, and screw the piston tightly. At the time, the total solution volume in No.1 flask should not exceed half of its volume.

Open No.7 pinchcock, close No.6 piston, heat No.8 flask, start to time when the solution in No.1 flask begins to boil, 2 minutes later, lower No.3 flask so that the lower end of the condenser tube exposes from the fluid level. Distil for 1 minute and wash the tube's lower end with water.

After the distillation is finished, tighten No. 7 pinchcock, cut off vapor, and let the solution in No.1 flask intake No.4 tube, loose No.7 pinchcock, add 40-50ml distilled water through No.5 funnel. And then add vapor to heat and back flow. Wash No.1 flask once for standby.

3.3 Titration

Titrate the absorption solution in No.3 titration flask with 0.01N hydrochloric acid solution until the solution appears light violet red.

In order to eliminate reagent error, perform a blank test by the operation method described above without adding test sample.

4 Calculation of Results

The dry basis content of crude protein is calculated as the following formula:

$$\text{Crude Protein (dry basis, \%)} = (V_1 - V_0) \times N \times 14 \times P \times \frac{50}{V} \times \frac{10000}{W(100 - M)}$$

Where: V-the dissolved digestion solution volume consumed in distillation, ml;

V_1-the hydrochloric acid solution volume consumed in the test, ml;

V_0-The hydrochloric acid solution volume consumed in the blank test, ml;

N-The equivalent concentration of hydrochloric acid solution, N;

14-Hydrochloric acid per milligram equivalent that is equivalent to the amount of azote;

P-Protein conversion factor (5.7 for wheat; 6.25 for other grains and beans);

W-The test sample weight, mg;

M-The percentage of moisture content of test sample, %.

The allowable deviation of the dual test results shall be no more than 0.2% when the crude protein content is below 15.0%, no more than 0.4% when the crude protein content is above 15.1%. Figure out their mean value and take the last digit after the decimal point as the result.

Notes:

? If the sulfuric acid loss is too much in the process of digestion, add sulfuric acid properly. Do not make the inner flask dry up.

? After the digestion solution is diluted with water, distill it in time, otherwise keep the digestion solution and dilute it just before use.

? The alkali liquor added into distillation No.1 distillation flask must be excessive.

? It is also advisable to use mixed indicator prepared with one portion of 0.2% methyl red ethanol solution and 5 portions of 0.2% bromocresol green ethanol solutions (mix just before using), it appears grayish-red finally.

Annex A

Methods for Determination of Soybean Water-soluble Protein

(Supplemental)

A 1 Instruments and Apparatus

A.1.1 Disintegrator;

A.1.2 Conical flask with ground stopper: 500ml;

A.1.3 Oscillator: international type;

A 1.4 Volumetric flask: 250ml;

A 1.5 Centrifugal machine, with 50ml centrifugal tube

A 1.6 Glass funnel;

A 1.7 Conical flask, pipette;

A 1.8 Other instruments and apparatus: same as those in the first chapter of this standard.

A 2 Reagent

The reagent used is the same with that in the second chapter of this Standard.

A 3 Operation Method

A 3 1 Preparation of test sample: porphyrize the soybeans so that 90% can pass through 60-mesh sieve.

A 3.2 Abstraction: Weigh up 5g porphyrized test sample (*W*, accurate to 0.01g), pour it into a conical flask with ground stopper, add 200ml water into the flask, and shake the flask to disperse the mixture evenly. And then oscillate it at a temperature of 25-30? for 2 hours, take it out and pour it into a 250ml volumetric flask, dilute it with water to the scale, mix evenly and keep it stand for 1-2 minutes, pour the upper clear solution into a centrifugal tube. Centrifuge it for 10 minutes in a centrifugal machine which rotates 1500 turns per minute. Filter the centrifuged material with quick filter paper or glass fiber and collect the clear filter liquor into a conical flask, i.e. the solution used to determine the water-soluble protein.

A 3.3 Nitrogen determination: Absorb and take 10ml test solution into 50ml or 100ml Kjeldahl Nitrogen Determination Flask. Digest, distil and titrate as per the procedures of 3.1, 3.2 and 3.3 of this standard. Note the amount of hydrochloric acid used in titrating the sample and blank solution.

A.4 Calculation of Results

The content of water-soluble crude protein is calculated as the following formula:

Water-soluble protein (dry basis, %)= $(V_1 - V_0) \times N \times 0.014 \times 6.25 \times 10/250 \times 5/50 \times 10000/W(100-M)$

Where: V_1-The hydrochloric acid volume consumed in titrating 5ml sample solution, ml

V_0-The hydrochloric acid volume consumed in titrating 5ml blank solution, ml

N-The equivalent concentration of hydrochloric acid solution, N;

0.014-Azote gramme per milligram equivalent;

6.25-The azote content of soybeans converted into protein coefficient;

10-The extraction solution volume absorbed for digestion, ml;

250-The volume of total extraction solution, ml;

5-The digestion volume absorbed for distillation, ml;

50-The volume of total digestion solution, ml;

W-The test sample weight, g;

M-The percentage of test sample's moisture content, %.

The allowable deviation of the dual test results shall not exceed 0.4%, figure out their mean value and take the first digit after the decimal point as the result.

Notes:

? The calculation method of dissoluble index (%) of soybean azote: Divide the measured soybean dissoluble protein (%) by the total soybean protein (%), and then multiplies it by 100.

? The sample preparation and extraction method make the determination result relatively stable. However, if the porphyrization fineness of the sample can't meet the requirements, then it is advisable to weigh 5.00g sample in the mortar, add 5ml water, and grind it by hand for 10 minutes. Transfer the intermixture with 200ml water into a 250ml ground flask. Shake it on the oscillator for 30 minutes, and then implement volume fixation, centrifugation, filtration and nitrogen determination as described in 3.2 of this standard.